

# COATING CHARACTERIZATION AND EVALUATION OF DIRECTIONALLY SOLIDIFIED CM 186 LC<sup>®</sup> AND SINGLE CRYSTAL CMSX-4<sup>®</sup>

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## ABSTRACT

Hot corrosion and long-time dynamic oxidation tests were conducted on several alloy (equiaxed MM-0011 and IN738, SX CMSX-4, and DS CM186LC) and coating (Simple aluminide, Pt-aluminide, and NiCoCrAlY) combinations. The coating performance in both hot corrosion and oxidation testing was, in most cases, influenced by the substrate composition. An overlay NiCoCrAlY on either CMSX-4 or CM186LC appeared unaffected visually, but there were indications during metallographic examination that this coating substrate combination was beginning to degrade after a 1000 hour hot corrosion test. The results of the oxidation test indicated that several of the alloy/coating combinations that were superior in the hot corrosion test were inferior in oxidation. After 6000 hours of dynamic oxidation testing at 1038°C (1900°F) two of the alloy/coating combinations had not yet reached negative weight changes. Metallographic results from the oxidation test show a significant reaction layer in the CMSX-4 coated with either simple or modified aluminides or NiCoCrAlY overlay coatings.

## NOMENCLATURE

PtAl Platinum aluminide coating  
AEP Allison electrophoretic process  
Aluminide Simple aluminide coating  
NiCoCrAlY an overlay nickel-cobalt-chromium-aluminum-yttrium coating  
HC short hand for hot corrosion test  
DO short hand for dynamic oxidation test  
CMSX-4<sup>®</sup> A single crystal alloy containing Re  
CM186LC<sup>®</sup> A directionally solidified alloy containing Re

## INTRODUCTION

Advances in turbomachinery have come about due to design and material improvements. Effective cooling schemes account for approximately 50% of the improvement while material process

advances account for the rest (Pickering 1986). The most significant process improvement can be attributed to the development of single crystal casting and composition technologies, with most efforts being pioneered by Pratt and Whitney (Gell et al. 1980, Harris et al. 1986)

Directionally solidified (DS) and single crystal (SX) alloys have differing compositions to account for the presence or absence of grain boundaries. The DS alloys contain elements such as carbon, boron, and zirconium for grain boundary strengthening. Hafnium is added to prevent hot tearing of the grain boundaries (Ross 1987) during solidification of components and to impart transverse strength during service. Rhenium is known to slow diffusion and is expected to have a beneficial effect on coating life.

Recent work has demonstrated the benefits of reducing the sulfur content on the oxidation resistance (McVay et al. 1992). Cannon Muskegon has taken this approach in their production heats of material and consistently produce product that has 1-2 wppm S (Harris 1994). The benefit of lower sulfur in the substrate has also been realized for coated CMSX-4 where a platinum aluminide coating performed better on an alloy with 1 wppm sulfur compared to a 6 wppm (Thomas et al. 1994)

This paper describes the results of hot corrosion and oxidation testing of bare and coated DS CM186LC and SX CMSX-4.

## PROCEDURE

As part of the Advanced Turbine System (ATS) design effort, Allison is conducting long term oxidation and hot corrosion tests of bare and coated CMSX-4 and CM186LC. The ATS program is intended to develop power generation gas turbine engines that have lives to 30,000 hours without shut-down while operating at or near peak temperature conditions. This aggressive goal must be achieved with conventional materials for the initial phases of the program. Follow on efforts incorporate higher risk non-metallic systems.

Allison Single Crystal Operations (SCO) cast tensile bars of CMSX-4 and CM186LC were subsequently solution heat treated to 99%, HIP and machined into cylindrical test specimens. The CMSX-4 bars came from a single low S heat of material as did the

Table I. Nominal compositions of alloys tested for this work.

Alloy	Al	Cr	Ti	Co	Mo	Ta	W	Re	Hf	Zr	Ni
IN738	3.45	16.0	3.45	8.5	1.75	1.75	2.6	—	—	0.055	Bal
MM-0011	5.5	8.4	1.05	10.0	0.65	3.05	10.0	—	1.4	0.055	Bal
CMSX-4	5.6	6.5	1.0	9.65	0.6	6.5	6.4	2.95	0.95	<0.0075	Bal
CM186LC	5.75	6.25	0.7	9.25	0.5	3.5	8.5	2.95	1.5	.007	Bal

CM186LC. The compositions of the alloys tested for this work are listed in Table I.

Samples were tested in the bare and coated conditions. The hot corrosion test accommodated a total of 28 samples while the dynamic oxidation test accommodated 12 samples in the carousel at one time. Samples were tested in DO by substitution after various intervals or conditions were reached. Table II lists the substrate and coating combinations that were tested in both the dynamic oxidation and hot corrosion tests. Several samples have been tested in the dynamic oxidation by substituting them in the test after a total of 5000 hours were completed on the original test pins.

The aluminide coatings were applied by an Allison licensed electrophoresis process (AEP). The NiCoCrAlY coating was applied by electron beam physical vapor deposition (EB-PVD) at a subcontractor in the US. In all cases the diffusion cycle corresponded to the primary age cycle.

Table II. Sample designation and coating substrate systems tested in this evaluation.

Sample	Test	Alloy	Coating type	Coating material
B1X	HC/DO	CMSX-4	None	None
N1X	HC/DO	CMSX-4	Overlay	NiCoCrAlY
P1X	HC/DO	CMSX-4	Diffusion	PtAl
S1X	HC/DO	CMSX-4	Diffusion	Aluminide
B2X	HC	CM186LC	None	None
N2X	HC	CM186LC	Overlay	NiCoCrAlY
P2X	HC/DO	CM186LC	Diffusion	PtAl
S2X	HC/DO	CM186LC	Diffusion	Aluminide
B5X	DO	CMSX-4La	None	None
P5X	DO	CMSX-4La	Diffusion	PtAl
S5X	DO	CMSX-4La	Diffusion	Aluminide
B8X	DO	IN738	None	None
N8X	DO	IN738	Overlay	NiCoCrAlY
P8X	DO	IN738	Diffusion	PtAl
B7X	DO	MM-0011	None	None

Note: B is Bare, N is NiCoCrAlY, P is PtAl, S is simple aluminide. The first digit identifies the substrate alloy and the second identifies the specimen number starting with "0".

The environmental tests, oxidation and hot corrosion, were conducted on two separate pieces of equipment rather than a combined test (Nelson 1995). Individual tests have been used

extensively as a screening method at Allison with good correlation to engine service.

The hot corrosion test was run at 899°C (1650°F) with 1% S (added as tertiary butyl disulfide) and 10 ppm synthetic sea salt aspirated into the flame. This test condition has been estimated to correspond to approximately a 10:1 ratio of test exposure to field exposure, with the test being nearly ten times more aggressive. Visual and metallographic analysis were used to determine sample failure which was defined as the formation of a blister (gross localized hot corrosion attack).

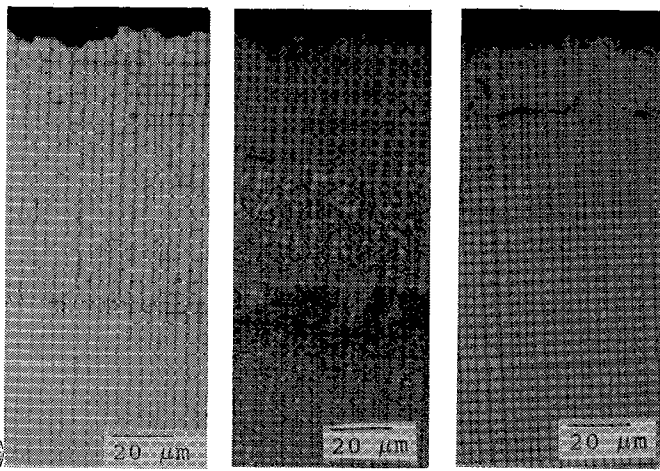
The dynamic oxidation test was conducted at 1038°C (1900°F) which was selected based on design considerations. A Becon burner rig was used. The Becon burner is a comprised of a modular combustor, which closely models the flow, mixing, and combustion chemistry of many industrial burners. Fuel, JP 5 in this case, is introduced into the front of the combustor and the combustion is stabilized by a strong swirl recirculation zone. The hot combustion gases are cooled by dilution by secondary air located in the back of the combustor. The samples rotated at 600 RPM and had a dwell time of 60 minutes in the flame and 5 minutes cold air blast. Sample failure was not predefined.

## RESULTS AND DISCUSSION

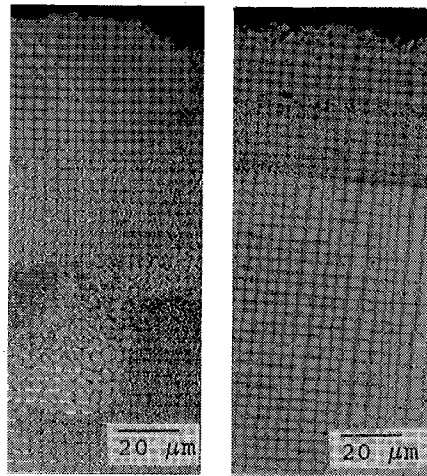
The original coating microstructures are shown in Fig. 1. The diffused aluminide coated samples show typical two layer structures. The outer layers are aluminum rich while the inner layers are rich in refractory elements. The AEP coating process has microstructural features generally associated with inward diffusion aluminide coatings. The two phase structure common to the NiCoCrAlY overlay coatings is evident in Fig. 1c. There appears to be more eutectic gamma prime in the CM186LC sample coated with AEP PtAl than in the simple aluminide coated one.

With the exception of IN738 which is a chromia former, the alloys and coatings tested for this evaluation are aluminum oxide (alumina) formers. Aluminum is selectively oxidized from the alloy or coating and forms a compact adherent oxide layer that decreases the diffusion of oxygen to the sample. As Al is oxidized, the ability for the alloy to form alumina is decreased. At some time, the alloy will be unable to form alumina and less protective oxide scales form. This leads to a second stage of oxidation in which the oxide has a higher growth rate and more tendency to spall during thermal cycling. The growth of chromia scales is similar to that for alumina scales except the rates are typically higher and the oxide is volatile above 1000°C.

There are three simple oxide growth behaviors that may be observed during cyclic oxidation: 1. Rapid increase in mass reflecting growth of non-protective mixed oxides, followed by a decrease in mass due to oxide spallation. 2. A rapid initial weight gain followed by slow, nearly zero mass change. This behavior is indicative of the formation of a protective oxide scale and is the preferred oxide growth morphology. And 3. a continuously decreasing mass change due to rapid oxide growth and spallation. These three behaviors can occur within one sample depending on the length of the test. Coatings, which have higher Al content than the base metal, can increase the length of time that an alloy remains in slow oxide growth regime.



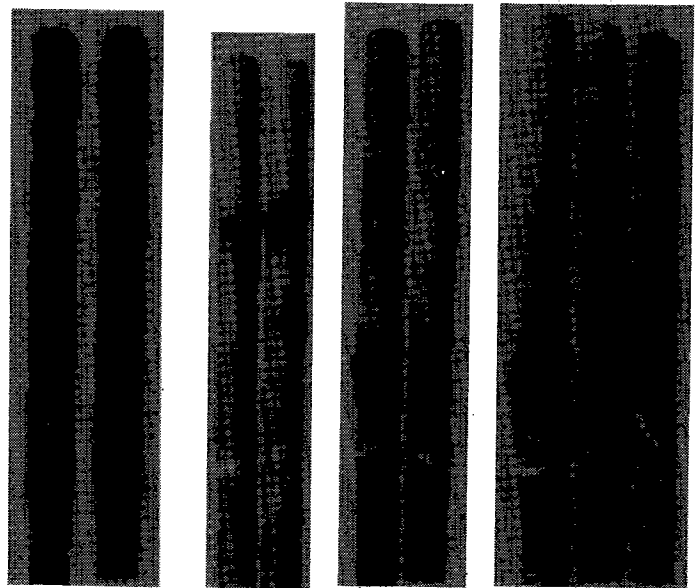
(a) (b) (c)



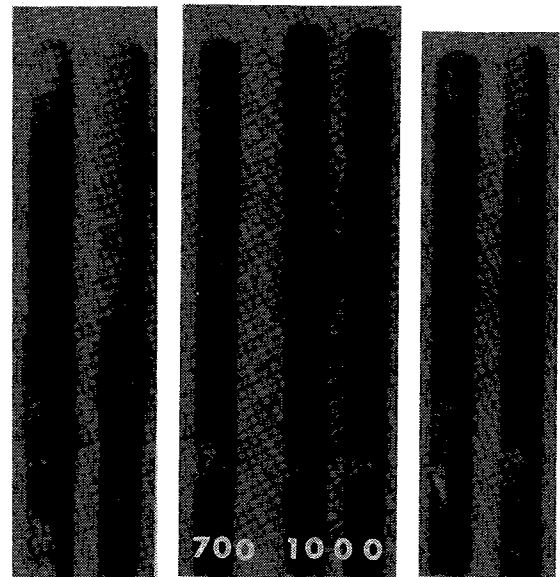
(d) (e)

Fig. 1. Original coating microstructures (a) CM186LC/NiCoCrAlY (b) CMSX-4/AEP PtAl (c) CMSX-4/AEP aluminide (d) CM186LC/AEP aluminide (e) CM186LC/AEP PtAl

The nominal coating thicknesses are listed in Table II. The simple aluminide has about the same thickness on both CMSX-4 and CM186LC (i.e. approximately 50μm). A similar correlation exists for the PtAl except the coating thickness is about 30% greater (approximately 72μm) on both alloys.



(a) (b) (c) (d)



(e) (f) (g)

Fig. 2. Surface condition of hot corrosion test pins after failure or test termination (a) CM186LC/50 hrs (b) CMSX-4/100 hrs (c) CM186LC/AEP aluminide/200 hrs (d) CM186LC/AEP PtAl/600 hrs (e) CMSX-4/AEP aluminide/600 hrs (f) CMSX-4/AEP PtAl/700 and 1000 hrs (g) CMSX-4/NiCoCrAlY/1000 hrs

### Hot Corrosion

The surface condition of the samples after failure occurred in the hot corrosion test are shown in Fig. 2. The bare CM186LC had poor hot corrosion resistance and lasted less than 24 hours since severe scaling was observed after this exposure interval. CMSX-4 on the other hand lasted nearly 100 hours prior to blister formation. Since diffusion coatings take on many of the chemical characteristics of the substrate one expects that the hot corrosion resistance of the CM186LC pins should be less than that of the CMSX-4, as was observed. The NiCoCrAlY coated pins showed no evidence of blistering after 1000 hours.

Table III. Nominal coating thickness of the samples tested

Sample Id.	Coating thickness ( $\mu\text{m}$ )	HC life (hours)
B1X	N/A	100
B2X	N/A	<24
S2X	50	160
S1X	56	522
P1X	71	740
P2X	74	730
N1X	76	>1000
N2X	74	>1000

The coating thicknesses are also indicated in Fig. 2. Contrary to expectation, the life improvement for the platinum aluminide coating on CMSX-4 can largely be attributed to its greater thickness. The increased life of PtAl over simple aluminide is essentially the same as its increased coating thickness (approximately 30%). On the other hand, a thickness increase of 40% (PtAl to simple aluminide) on CM186LC produced a life improvement of 350%.

Figure 3 shows the typical coating and substrate microstructures after sample failure or in the case of NiCoCrAlY after the test was terminated. There is a significant loss of parent metal in both bare samples. The losses for the coatings are related to the substrate and coating. The aluminide coatings exhibited localized attack of both samples. The NiCoCrAlY coating on CMSX-4 did not have any areas of attack; on the other hand, the NiCoCrAlY coated CM186LC typically had a 5 $\mu\text{m}$  depletion near the surface. In addition to the general attack, there was one area that had been fully denuded of beta phase. Hot corrosion behavior is a complex subject in which many coating and substrate elements play an important role. Rapp and Zhang (1994) describe hot corrosion based on fundamentals and should be read for a more detailed discussion. The simplest phenomenology for hot corrosion is one in which the oxides are removed due to liquation and subsequent fluxing of the protective oxide scales. Various elements, such as W and Mo, promote acidic fluxing of the oxide scale. Hafnium may also play an important role in allowing a short circuit diffusion path for sulfur containing reactants (Hwang et al. 1983).

The lives of the bare and coated CMSX-4 and CM186LC are shown in Fig. 4. The NiCoCrAlY coated pin did not fail after 1000 hours of exposure. This corresponds to a life of approximately 10,000 hours or over one third of the desired lifetime for a hot corrosion environment only.

### Dynamic Oxidation

The dynamic oxidation (DO) behavior of the alloys and coating listed in Table I was evaluated. The condition of the bare alloys after the exposure times indicated are shown in Fig. 5. The samples show varying degrees of distress depending on the alloy and exposure time. Sample B10 (CMSX-4) shows a significant diameter reduction. This result is in agreement with the specific mass change data. The mass loss accelerated after 1000 hours.

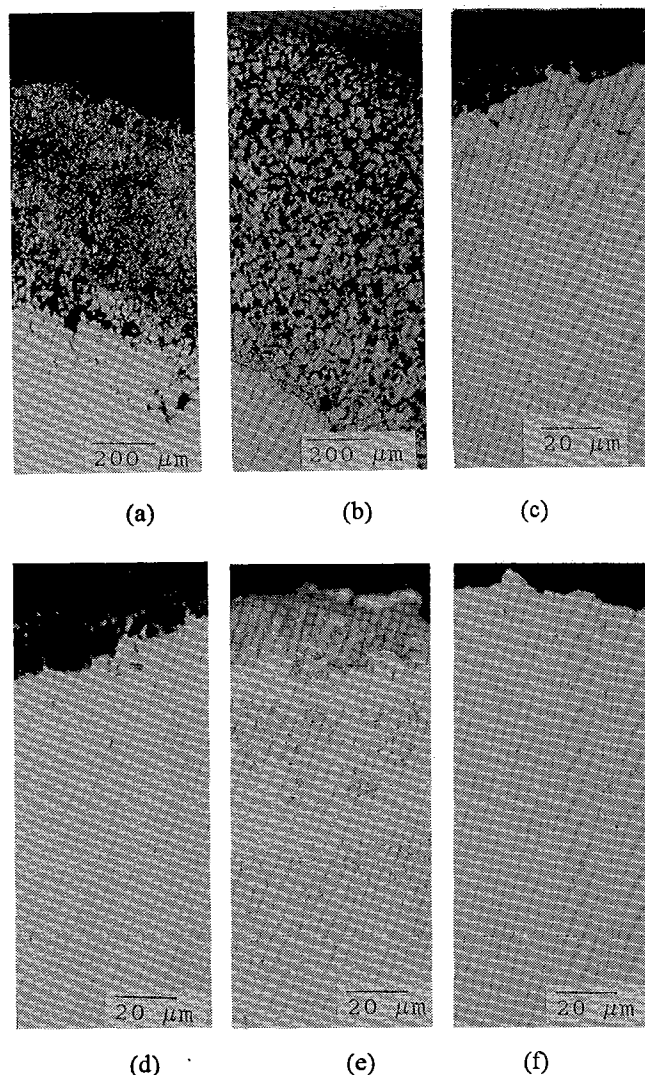


Fig.3. Sample microstructures after hot corrosion failure due to blister formation (a) Bare CM186LC/50 hrs (b) Bare CMSX-4 200 hrs (c) CMSX-4/AEP Aluminide/600 hrs (d) CM186LC/AEP PtAl/600 hrs (e) CMSX-4/AEP PtAl/700 hrs and (f) CM186LC/NiCoCrAlY/1000 hrs

Sample B81 (IN738) shows a noticeable diameter reduction after only 545 hours. The diameter loss is calculated (based on density and mass loss) to be approximately 0.152 mm. No distress is apparent for samples B51 (CMSX-4+La) and B71 (MM-0011) after

970 and 425 hours exposure, respectively, indicating that the oxide films that are forming are still protective.

The gravimetric data for the bare alloys tested to 1000 hours is shown in Fig. 5b. Based on these data, it can be seen that the bare IN738 reached a mass loss of 20 mg/cm<sup>2</sup> (which corresponds to approximately 50 μm surface loss) after approximately 320 hours at 1038°C. The life of CMSX-4 to 50 μm loss would be approximately 1300 hours. MM-0011 and CMSX-4+La showed little or no mass loss and the expected life to 50 μm suraceloss would be well beyond 1000 hours. These data clearly show the benefit of the La addition on the oxidation behavior of CMSX-4.

The comparative specific weight change after 1000 hours is -6 mg/cm<sup>2</sup> for CMSX-4 and +0.25 mg/cm<sup>2</sup> for CMSX-4 + La. Reactive element doping has been demonstrated to improve the oxidation characteristics of a number of nickel based alloys and coatings. CMSX-4 + 15 ppmw Y showed a dramatic improvement over conventional CMSX-4 when tested at 2150°F (Thomas et al. 1994). It has been postulated that the rare earth additions, in particular Y but presumably also La, improve oxide adherence by tying up sulfur as oxysulfides (Almone and McCormick 1992).

Based on the previous discussion of desirable oxidation traits, CMSX-4 + La has the best oxidation resistance of the materials tested. The oxidation behavior of the IN738 is not unexpected since chromium oxides form a significant fraction of gaseous oxides above 1000°C. The gradual mass of MM-0011, although not ideal, is preferred to the rapid mass loss of the IN738 sample.

The surface condition of the bare and coated CMSX-4 + La samples after approximately 1000 hours exposure at 1038°C is shown in Fig. 6a. No distress of either the bare or coated samples is noted.

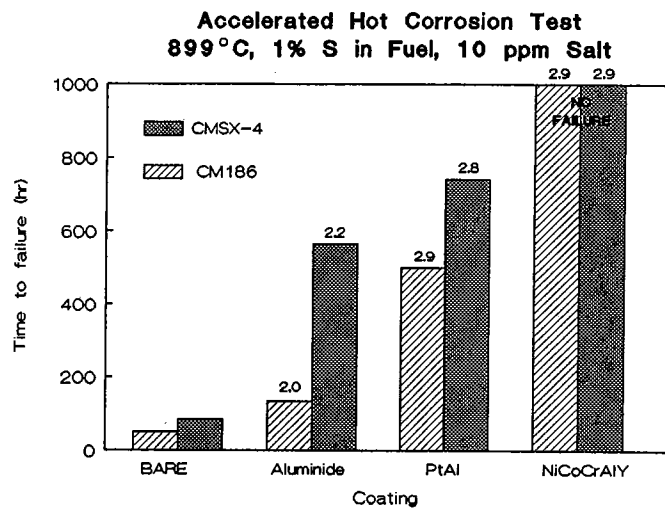
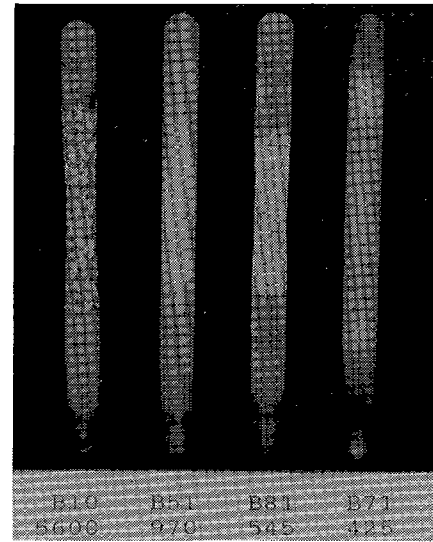


Fig. 4. Life of bare and coated CMSX-4 and CM186LC in Type I hot corrosion testing.

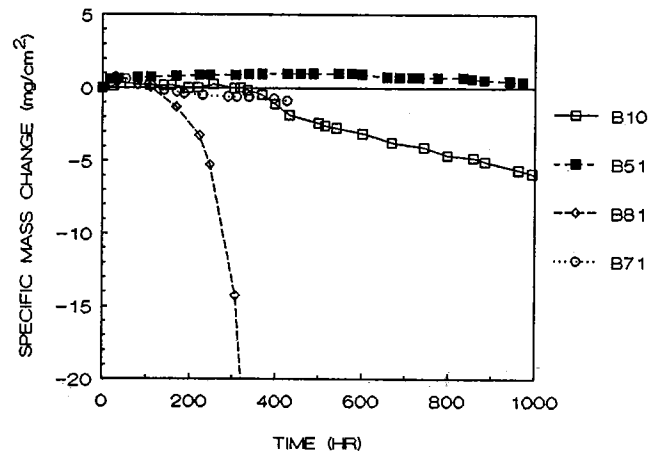
The gravimetric results for the CMSX-4 + La are shown in Figure 6b. These data all show a positive mass changes after 1000 hours but the bare sample has started to lose mass due to oxide spallation. There is a sizable difference in the initial weight gains

for alloys and coatings which form predominately Al<sub>2</sub>O<sub>3</sub> as a protective oxide. High initial mass gains are associated with thicker oxide layers or the formation of more transient oxide films. These thicker transient oxide layers can promote oxide spallation at shorter times.



(a)

**DYNAMIC OXIDATION 1038°C  
CMSX-4, CMSX-4+La, IN738, MM-0011**

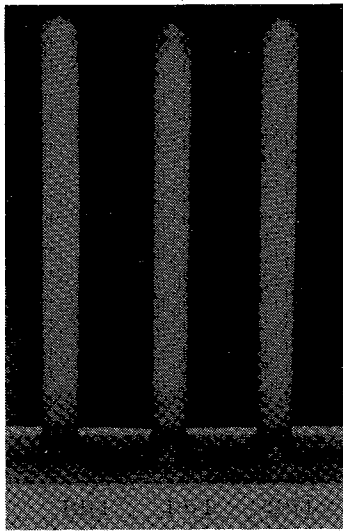


(b)

Fig. 5. (a) Surface condition and (b) gravimetric results for bare alloys CMSX-4, CMSX-4+La, IN738, and MarM 247, after dynamic oxidation testing at 1038°C for indicated times.

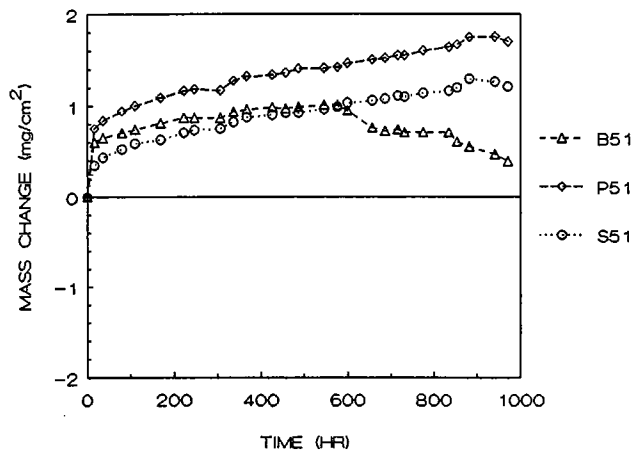
The surface condition of the coated CMSX-4 and CM186LC samples after approximately 6000 hours exposure at 1038°C is shown in Fig. 7. Localized attack is evident on sample S20; while not visible in this view, a larger area has been oxidized and eroded away on the "back" side of S10. These localized areas of oxidation account, in part, for the mass losses experienced by these two samples. No localized attack or diameter loss is apparent for the

CMSX-4 coated with either AEP platinum aluminide or NiCoCrAlY overlay or for CM186LC coated with AEP PtAl.



(a)

**DYNAMIC OXIDATION 1038°C CMSX-4 + La**  
Bare, AEP PtAl, AEP Aluminide

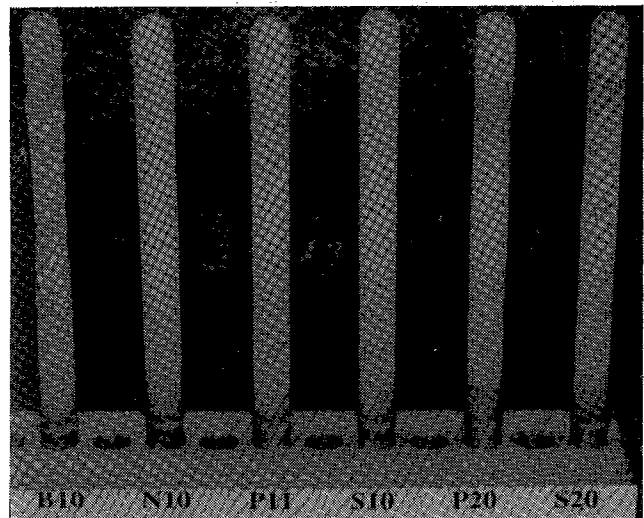


(b)

Fig. 6. (a) Surface condition and (b) gravimetric results for bare and coated CMSX-4+La after dynamic oxidation testing at 1038°C for 970 hours.

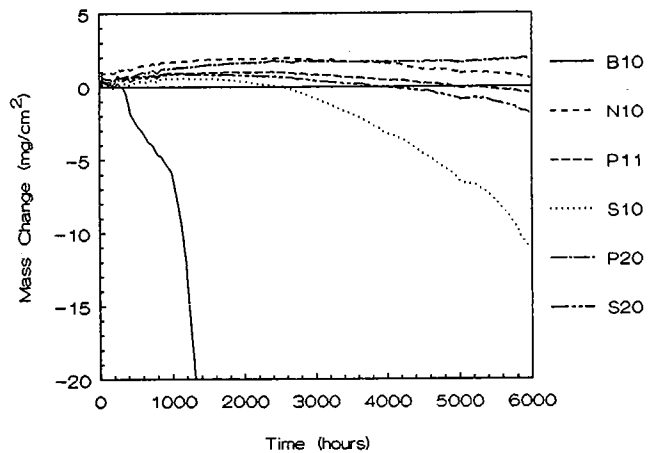
The gravimetric results for bare and coated CMSX-4 and coated CM186LC are shown in Figure 7b. The only sample that has reached the arbitrary limit of 20 mg/cm<sup>2</sup> (50μm) is the bare CMSX-4. The other samples have not lost 20mg/cm<sup>2</sup> after nearly 6000 hours exposure at 1038°C. The weight change data for the bare and coated samples are consistent with the coating and substrate compositions. For coatings on CMSX-4, the simple aluminide demonstrated a negative weight loss first, followed by the platinum aluminide. A similar behavior is evident for the CM186LC, with the simple aluminide showing negative weight loss after

approximately 4200 hours and the PtAl not yet reaching its peak. The specific mass change of the NiCoCrAlY on CMSX-4 has not reached negative values yet but it is clear that a fully protective alumina scale is no longer being formed. The superior oxidation resistance of the coated CM186LC can be attributed, in part, to the Hf which is added as a grain boundary strengthener and to prevent hot tearing. The addition of Hf has also shown improved oxidation resistance (Allam et al 1978).



(a)

**Dynamic Oxidation 1038°C**  
Bare and Coated CMSX-4 and Coated CM186



(b)

Fig. 7. (a) Surface condition and (b) gravimetric results for bare and coated CMSX-4 and coated CM186LC after dynamic oxidation testing at 1038°C for nearly 6000 hours.

Samples were removed from the DO test after various times. These samples were sectioned and evaluated metallographically.

Typical microstructures are shown in Figures 8 and 9 for samples exposed 1000 and 3500 hours respectively. The bare CMSX-4 samples exhibited voids near the surface. These have been attributed to vacancy coalescence (McVay et al. 1992). The surface roughness and mass losses can likely be attributed to the growth and coalescence of the voids. The erosion effect of the high velocity gas stream also contributes to the weight loss and surface roughness.

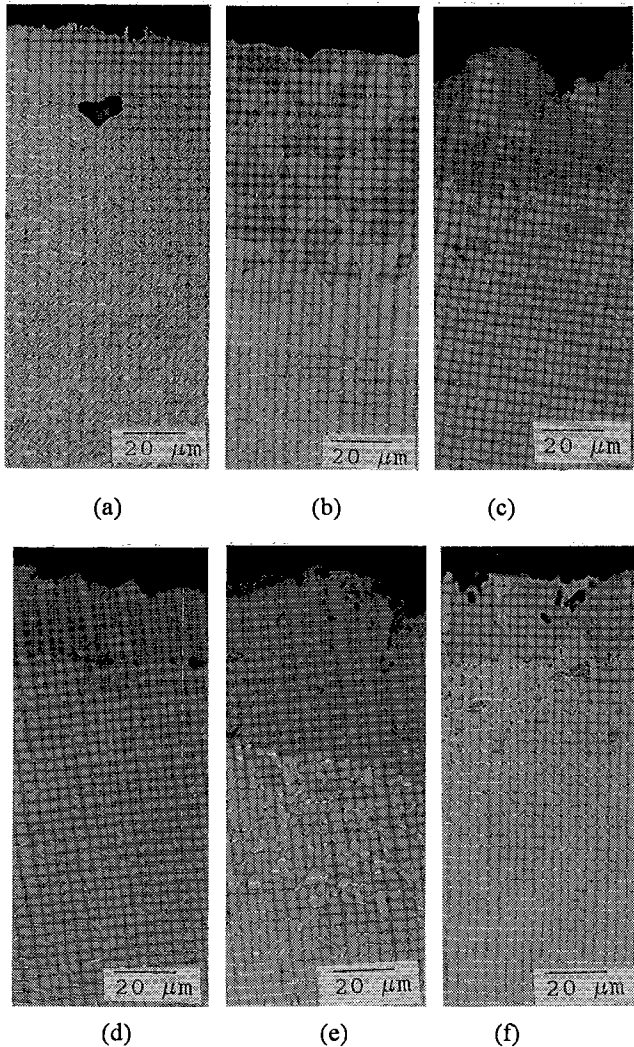


Fig. 8. Typical microstructures of bare and coated CMSX-4 and coated CM186LC after 1000 hours of dynamic oxidation testing at 1038°C, (a) Bare CMSX-4 (b) CMSX-4/NiCoCrAlY (c) CMSX-4/AEP PtAl (d) CMSX-4/AEP Aluminide (e) CM186LC/AEP PtAl (f) CM186LC/AEP aluminide

The microstructure of the coated samples after 1000 hours exposure, Fig. 8, show a number of features associated with thermal exposure. The beta phase of the NiCoCrAlY has coarsened significantly from the original size shown in Fig. 1. There is also evidence of coarsening in the four aluminide coated samples. In addition to the coarsening, some second phases have precipitated beneath the coatings. Depending on the precipitate morphology, the

presence of these precipitates can have a detrimental effect on the rupture life.

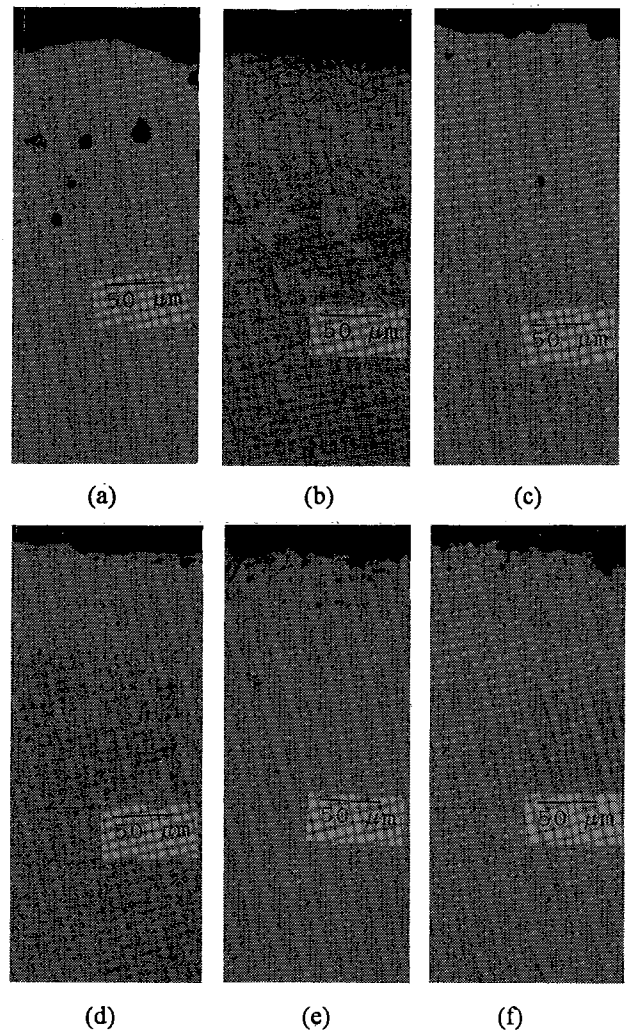


Fig. 9. Typical microstructures of bare and coated CMSX-4 and coated CM186LC after 3500 hours of dynamic oxidation testing at 1038°C, (a) bare CMSX-4 (b) CMSX-4/NiCoCrAlY (c) CMSX-4/AEP PtAl (d) CMSX-4/AEP Aluminide (e) CM186LC/AEP PtAl (f) CM186LC/AEP aluminide

Figure 9 shows that more degradation has occurred in the coated samples. There are more precipitates present in all of the coated samples. The beta phase etches like the coating matrix in the CMSX-4/NiCoCrAlY sample, Fig 9b. The beta phase enriched zone of the AEP PtAl coated CMSX-4 does not etch as darkly as it originally did indicating a depletion in the Al content. There is no beta phase enriched layer visible in the aluminide coated CMSX-4, Fig 9d. This observation is consistent with the mass loss which indicated a metal loss on the order of 25 μm. The total coating thickness was 56μm, of which approximately 20 μm was the beta phase enriched zone, Fig 1d. The CM186LC sample has internal

oxide "pegs" visible near the surface. These pegs occur on materials that contain significant amounts of reactive elements such as Hf. The arrows highlight two such precipitates. No needle-like phases are evident in the AEP PtAl coated CM186LC sample, Fig. 9e. The microstructure of the AEP aluminide coated CM186LC, Fig. 9f, shows very little if any beta phase enriched layer. There appear to be some oxide intrusions but not to the extent shown in the PtAl coated CM186LC. Needle like TCP phases are evident and extend approximately 250  $\mu\text{m}$  from the surface.

## CONCLUSIONS

The hot corrosion behavior of bare CM186LC is worse than bare CMSX-4; this is likely due to the presence of more W and Hf in CM186LC.

There was little benefit for hot corrosion resistance realized in using PtAl on CMSX-4 compared to a simple aluminide. The benefit in life could be attributed to differences in coating thickness.

The oxidation life of bare IN738 (chromia former) is significantly shorter than the  $\text{Al}_2\text{O}_3$ -forming alloys.

A dramatic improvement in life was demonstrated by low dopant levels of La in CMSX-4.

The oxidation life of PtAl coated CM186LC was superior to the other materials dynamically oxidation tested at 1038°C.

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